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(54) Title of the Invention:
METHOD TO DECREASE OXYGEN IN RARE EARTH METAL

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SPECIFICATION

1. **Title of the Invention**

METHOD TO DECREASE OXYGEN IN RARE EARTH METAL

2. **What Is Claimed Is:**

Claim 1

A method of decreasing oxygen in rare metals, wherein a purification process is conducted in vacuum by using a container whose surface comprises at least an anticorrosive material formed by mixing one or more of Al_2O_3, MgO, and CaO in an amount of no more than 20 mole% into Y[illegible]O[illegible] or Y[illegible]O[illegible].

Claim 2

The method of Claim 1, wherein said container is made by forming a film of said material on the surface of a general material by plasma spraying.

3. **Detailed Explanation of the Invention**

Industrial Fields of Application

The present invention relates to a method to decrease oxygen in rare earth metals by using a container, wherein the surface of said container comprises an anticorrosive material.

Prior Art

Rare earth metals are important raw materials, from which various new materials, such as a magnetic material and special alloy, are derived. In applications utilizing such new materials, minute impurities frequently impair the quality of the materials significantly, and therefore, consideration must be given to the prevention of contamination during manufacturing and treating processes involving rare earth metals or alloy thereof. For example, rare earth metals are heated and melted in vacuum or in the atmosphere of very pure inert gas. However, there is a problem in that rare earth metals are highly active so that the rare earth metals react with the material of a

crucible during melting and molding processes thereof, which causes the corrosion of the crucible as well as the contamination of the rare earth metals per se.

For melting such metals that are readily subject to corrosion, conventionally materials that are resistant to reduction such as MgO, CaO, and Al_2O_3 have been used.

Problems to Be Solved by the Invention

However, even by using such metals, the prevention of the corrosion by a rare earth metal and the like is not sufficient. Because oxygen is incorporated into a metal being purified or melted, a product of good quality cannot be obtained.

To solve the problems mentioned above, the invention below has been concieved.

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The Constitution of the Invention

Thus, the present invention relates to:

(1) a method of decreasing oxygen in rare metals, wherein a purification process is conducted in vacuum by using a container whose surface comprises at least an anticorrosive material formed by mixing one or more of Al_2O_3, MgO, and CaO in an amount of no more than 20 mole% into Y[illegible]O[illegible] or Y[illegible]O[illegible]; and

(2) the method of (1), wherein said container is made by forming a film of said material on the surface of a general material by plasma spraying.

Means for Solving the Problems and Actions

Hereafter, the present invention is explained.

The metals that are subjected to the present invention include rare earth metals, and alloys (including Mischmetall), the principal component being rare earth metals.

To solve the problems mentioned above, we use Y[illegible]O[illegible] as a material having an anticorrosive property given to rare earth metals. An oxide of a rare earth metal, especially Y[illegible]O[illegible], is an oxide of an element having a strong affinity for oxygen, and hence the reactivity with rare earth metals is minimal. The anticorrosive material is solely used to form a container for treating active metals; or a film thereof is formed on the surface of a container made of a general inexpensive material that can be readily molded, such as Al_2O_3 and MgO, in turn giving the anticorrosive property to the material of the container. Such film forming method is useful especially when an expensive material such as Y[illegible]O[illegible] is used.

A material formed by mixing one or more of Al_2O_3, MgO, and CaO in an amount of no more than 20 mole% into Y[illegible]O[illegible] or Y[illegible]O[illegible] can be used as an anticorrosive material. Of oxides of rare earth metals, Y[illegible]O[illegible] is exclusively selected as an anticorrosive material, because a crystal structure changes between a melting point and ambient temperature for other oxides of rare earth metals, and also Y[illegible]O[illegible] has the least reactivity. It is desirable to add Al_2O_3, MgO, and CaO to Y[illegible]O[illegible], as

mentioned above, so that improvements for sintering, in adhesion to a underlying material on which a film is formed, for tight contact, and for suitable thermal expansion can be attained. However, these oxides are inferior in anticorrosion, and when added in a large amount, the anticorrosive property of the material itself is compromised exceedingly, so that the amount of such oxides added has to be 20 mol% or less. Also, there are other oxides that improve a sintering property than these oxides, but they are not suitable in terms of their anticorrosive property and prices.

An example wherein a container is solely made of the material by sintering is explained in detail below.

With sintering, fine powder with an average particle diameter of 10 μm or less, preferably 1μm or less, may be used to make a fine sinter. The powder is molded into a shape by compression such as through the application of pressure, which is the most typical method, or by cold isostatic press. A sintering temperature varies with the kinds and amounts of additives to mix with an oxide of a rare earth metal, but should be at least 1,400℃ or higher, preferably 1,700 ℃ or higher.

Also, forming a film is explained below.

There may be several methods for forming a ceramic film including: (1) sputtering; (2) CVD; (3) sintering after application of slurry; and (4) plasma spraying. Of these methods, however, the methods of (1), (2), and (3) pose problems such that a thick, strong film is difficult to form; the formation of a film on the inner surface of a crucible is difficult; a film formation rate is slow; and the strength of slurry adhesion is not sufficient.

In the present invention, a method is used, wherein a film of the anticorrosive material mentioned above is formed by plasma spraying on the inner surface of a crucible made of a general ceramic material having a relatively good thermal insulation such as alumina. In this case, advantages gained by using plasma spraying are: (1) a film can be made even on the surface of a container wherein the surface has complicated contours up to some extent; (2) upsizing is possible; (3) a film formation rate is fast; (4) a sufficiently thick film can be formed; (5) the strength of slurry adhesion is great; (6) the application on a ceramic material having a high melting point is

possible, etc.

Powder suitable for plasma spraying is nearly spherical; has an average particle diameter of smaller than 100 μm, preferably 50 to 5 μm; and has a good flowability. Any material that can resist to plasma spraying, heat used to treat active metals, and heat shock may be used for the underlying substrate on which plasma spraying is performed, but a material that can withstand to some extent the action of the active metals may be used for the substrate, in case that the anticorrosive film is peeled off. Suitable examples of such materials are Al_2O_3 and MgO. A film thickness is suitably 50 μm to 1 mm, more suitably 200 to 300 μm. With a thickness of smaller than 50 μm, the film strength is not sufficient, and with a thickness of larger than 1 mm, it takes exceedingly long to form such film, and hence forming a film having a thickness in these ranges is impractical.

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<Example 1>

To Y[illegible]O[illegible] with an average diameter of 4 μm, 4 wt% (about 8 mole%) of Al_2O_3 powder was added, which then was sintered at 1,700℃ for 3 hours. The obtained sintered object was a cylindrical crucible with a diameter of 30 mm having a [illegible] density of 99% (d = 4.87). In this crucible, Mischmetall was melted in vacuum. As shown in the table, an increase in oxygen incorporated into the material was small.

<Example 2>

A film of Y[illegible]O[illegible] with a thickness of 0.2 mm was formed by Ar plasma spraying on the inner surface of a cylindrical crucible made of MgO with an inner diameter of 35 mm. In this crucible, Mischmetall was melted in vacuum. As shown in the table, an increase in oxygen incorporated into the material was small.

<Reference Example>

In cylindrical crucibles with an inner diameter of 30 to 35 mm made of MgO and Al_2O_3, respectively, Mischmetall was melted in vacuum. As shown in the table, an increase in oxygen incorporated into the material was large.

Table 1: Comparison of an amount of oxygen [illegible] contained in Mischmetall after treating with heat in vacuum in a crucible (after heating at 1,100℃ for 1 hour).

Item	Subject	Oxygen contained in the material (ppm)
	The material / Mischmetall	90
Example 1	Y[illegible]O[illegible]-Al_2O_3	240
Example 2	Y[illegible]O[illegible], plasma sprayed	210
Reference Example	MgO	900
Reference Example	Al_2O_3	1,900

<u>Operation of the Invention</u>

(1) In the present invention, oxygen can be prevented from being incorporated into a rare earth metal during a melting or purifying process of such metal.

(2) The above effect of (1) can be obtained at low cost by forming a film.

(3) For example, when a metal containing a low level of oxygen treated in accordance with the present invention is used for making a hydrogen [illegible] alloy of rare earth metals, the generation of water is suppressed and satisfactory properties are obtained.

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TRANSLATOR CERTIFICATION

I, Megumi Nozawa, a translator fluent in the Japanese language, on behalf of Morningside Evaluations and Consulting, do solemnly and sincerely declare that the following is, to the best of my knowledge and belief, a true and correct translation of the document(s) listed below in a form that best reflects the intention and meaning of the original text.

MORNINGSIDE EVALUATIONS AND CONSULTING

Signature of Translator

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